THE NICKEL BULLETIN

A SUMMARY OF CURRENT INFORMATION ON NICKEL

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VOLUME 27 NUMBER 4

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THE IMPACT STRENGTH OF SPHEROIDAL-GRAPHITE CAST IRON

CAST IRON has many unique qualities as an engineering material, but one of its main deficiencies is its brittleness and lack of ductility, which limit its usefulness for service under conditions involving shock loading. However, although brittle in comparison with many other metals, the toughness of cast iron (the converse of brittleness) is variable, and some grades of iron castings have long been recognized as tougher than others. For example, castings made from cold blast pig iron have a name for toughness, and high phosphorus in the composition is known to make the castings especially brittle. Alloy additions such as nickel and molybdenum, when made to a suitable base iron, increase the toughness of the castings.

Wartime interest in the use of iron castings to replace certain steel forgings—then in short supply, and again the failure of iron castings under explosive shock especially at sea, both focussed attention on the relative degree of toughness of various grades of plain and alloy cast iron. No test for measuring the toughness of cast iron was available up to that time. The Izod test (B.S.131) standard for steel giving impact strengths in the range 10 to 100 ft. lbs., is unsuitable for cast iron as the latter gives figures of 1 ft. lb. or less and is thus incapable of discriminating satisfactorily between different grades. This problem was studied[1] and led to the publication of B.S.1349 covering an impact test in the Izod machine using a machined bar 0·798-in. in diameter and unnotched. Whilst this test has served a useful purpose it has its limitations, and for research purposes the Charpy test is preferred. The latter test is, incidentally, in general use abroad for assessing the impact values of both steel and cast iron.

The recent development of spheroidal-graphite cast iron, in which the iron as-cast contains the free graphite in the form of dispersed spheroids instead of in the normal sharp edge flake form led at once, as was expected, to a marked increase in the mechanical properties of iron castings, and in particular in their strength and toughness. S.G. iron is commercially produced in two main types, namely with a pearlitic matrix, as-cast, or with a ferritic matrix developed by a short time annealing (*see* Mond Nickel Co., publication 630, 'Spheroidal-Graphite Cast Iron'). The toughness of S.G. iron as-cast is approximately four times that of the corresponding flake graphite iron, whilst, in the annealed condition, the toughness is increased to about twelve times. However, it was not generally realised that the composition and especially the silicon content were vitally important and for that reason it was sometimes found that the product did not show the marked increase in toughness which was expected. Thus there are early reports of annealed S.G. iron which was quite satisfactory when subject to a slow bend test, as in a vice, but which fractured when dropped on to a hard floor. Further, it was noticed that the fracture of S.G. iron which came up to expectations showed a tough dull grey appearance, but in the anomalous cases met, the fracture was light in colour and appeared crystalline.

Since more precise data were required on this question an extensive research was undertaken in the laboratories of The Mond Nickel Company to determine the factors underlying these differences in behaviour. The results of this research have recently been published[2].

Initial work in this field showed that as in the case of steels, cast iron demonstrated a characteristic known as the impact transition temperature range. This is illustrated in Fig. 1 in which it will be seen that the casting shows at low temperature a relatively low impact strength, but over a temperature range which is characteristic of the composition and microstructure of the material, the impact strength rapidly increases to a higher level which is subsequently maintained as temperature rises further. It has been observed that the so-called brittle fracture showing a light crystalline appearance is characteristic of the low temperature, low impact, whereas the tough fracture with its dark grey fibrous appearance is

(1) A. B. EVEREST, J. W. GRANT & H. MORROGH: 'The Development of a Single-Blow Impact Test for Cast Iron.' *Jnl. Iron & Steel Inst.*, 1945, vol. 152, pp. 403–23.

(2) A. L. CARR & W. STEVEN: 'Impact Properties of Annealed Spheroidal-Graphite Cast Iron.' *Metal Treatment*, 1953, vol. 20, pp. 455–61. Obtainable as Mond Nickel Co. publn. 716.

characteristic of the high temperature, high impact condition. Mixed fractures are obtained within the limits of the transition range. The outstanding toughness of S.G. iron is associated with the fully fibrous fracture.

The present research aimed to relate composition with the impact transition temperature range. This clearly was vital since where toughness is required, the impact transition temperature range must be wholly below the operating temperature of the castings. The research was concerned mainly with the study of the influence of variations of the elements normally present, on the impact transition temperature range of annealed S.G. iron. Full details of the results are given in the paper by Carr and Steven.

The essential conclusions are as follows:—

(*a*) Silicon has very important effects on impact properties. It raises the impact transition range markedly and reduces the impact values obtainable at temperatures above the transition range, i.e., the impact strength associated with fully fibrous fracture is decreased by increase of silicon content. Raising silicon in the range 1·4 to 4·6 raised the transition range by more than 200°C. and when silicon exceeded about 3 per cent., the transition was mainly above room temperature, and at room temperature the castings had poor notched and unnotched impact properties, showing brittle fractures. These effects are illustrated by Fig. 1. Accordingly this part of the work leads to the conclusion that for maximum toughness in annealed S.G. iron, silicon should be kept low, and following experience over the past few years, it is recommended that silicon should be less than 2·5 per cent., except in special cases, for example when higher silicon is desired to give improved resistance to oxidation.

The influence of the other elements in cast iron has also been studied. Briefly it may be stated that manganese has an effect similar to that of silicon, though to a less marked degree. Provided silicon is adjusted to the level indicated above, normal variation of manganese is not likely to have a deleterious effect.

Phosphorus in S.G. iron is normally low, usually less than 0·10 per cent. Studies in this research were, however, extended to phosphorus levels up to 0·6 per cent. and they showed that phosphorus embrittles S.G. iron, as it does other types of cast iron, and should not exceed 0·15 per cent. if toughness is to be unimpaired. It was interesting to note that a marked deterioration in impact strength did not coincide with the first appearance in the microstructure of phosphide eutectic but coincided approximately with the development of networks of phosphide.

The effect of small proportions of residual pearlite in the annealed ferritic irons was also studied and it was found that pearlite resulting from incomplete annealing gave lower and more erratic impact strength than in the case of the fully annealed irons.

There is no inherent difficulty in producing

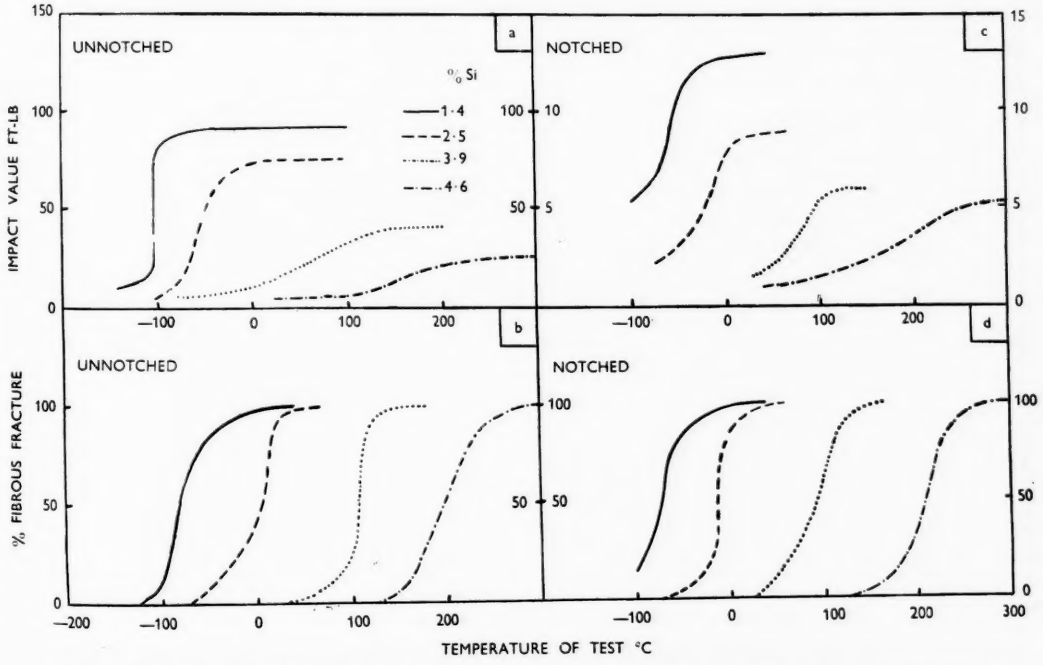


Fig. 1 The Influence of Silicon on the Impact Properties of Annealed Spheroidal-Graphite Cast Iron.

S.G. iron castings with high toughness levels, but where maximum resistance to impact is required, the composition must be controlled on the lines indicated above.

In the research under consideration, comparison was made also with high grade Blackheart malleable cast iron. The silicon content in the latter was in the range 1·0–1·2 per cent., that is substantially less than in S.G. iron. Consequently in such malleable cast iron, the impact transition is at a low temperature, and the toughness of such malleable iron lies generally at a somewhat higher level than for S.G. iron. The latter is usually harder than the malleable iron and consequently has higher impact transition range and lower maximum impact value. If, however, S.G. irons are specially made of the same hardness as malleable irons, then the S.G. irons have better impact properties.

In the research, tests were carried out on 10 mm. square test pieces, both notched and unnotched. A large number of results were obtained on which the above conclusions have been drawn. The tests were carried out in the Charpy machine mainly because that test is more suitable for testing specimens at high and low temperature.

This work confirms that the toughness of S.G. iron, which is several times that of the flake graphite types of iron, is of the same order as that of malleable cast iron, and, with suitable composition, is appreciably higher. The relationship between composition and impact transition temperature adequately explains the anomalous behaviour of some of the early heats of S.G. iron, and shows that suitable attention to composition control ensures the development of the desired toughness.

The increased toughness of S.G. iron opens up a wide field for the use of iron castings under conditions where shock is likely to be experienced. It is already replacing many parts previously made in steel with marked economy in production costs. In this connexion it is significant to note that in the U.S. a specification has been issued covering the use of S.G. iron for castings for use on board ship (U.S. MIL.I.17166A (Ships)).

Many of the applications of S.G. iron listed and illustrated in our publications depend for their success on the increased toughness shown by the new metal.

NI-RESIST

NI-RESIST is the name given to a group of high-nickel, austenitic cast irons. These irons were developed to provide maximum resistance to corrosion and heat, and they bear somewhat the same relationship to ordinary cast irons as do the austenitic stainless steels to ordinary steels.

In consequence of its composition and austenitic structure Ni-Resist has special mechanical and physical properties. It is much tougher than ordinary cast iron, and also has good resistance to wear and erosion. Both the types referred to below are non-magnetic; they have high thermal expansion and high electrical resistivity. The damping capacity and modulus of elasticity are similar to those of ordinary cast iron such as B.S. 1452, grade 12.

GRADES

The normal range covers two grades, namely:—

'Standard' Ni-Resist: Recommended for general corrosion- and wear-resistant applications. e.g., pipes, valves, pumps, cylinder liners, etc., and for heat-resisting items operating at temperatures up to 800°C.

'Copper-free' Ni-Resist: As above, but preferred for use in the handling of caustic liquors, ammoniacal solutions, for the processing of foodstuffs and for all applications in which freedom from copper contamination is essential. In other respects it is similar to 'Standard' Ni-Resist. Details of composition and properties will be found in Table I, p. 60.

Further modifications of composition have been developed to meet specific needs such as low thermal expansion and extra resistance to thermal shock, staining, higher temperatures or severe abrasion.

The here-mentioned irons are all of the flake graphite type. A marked increase in strength, ductility and toughness may be obtained without sacrifice of other properties by magnesium treatment to give Ni-Resist in which the graphite is spheroidal.*

* See *Nickel Bulletin*, vol. 26 1953, p. 34. Reprinted as Publication No. 637.

CORROSION-RESISTANCE

The outstanding property of Ni–Resist is its resistance to attack by many common corrosive agents and in particular by sea water, weak acids and alkalis. Typical data are given in Table II on p. 61 in which the corrosion rate of Ni–Resist is compared with ordinary cast iron and phosphor bronze in several corrosive media.

HEAT-RESISTANCE

Due to its stable austenitic structure and high alloy contents, Ni–Resist is many times more resistant to growth and oxidation than ordinary cast iron. It retains much of its strength at elevated temperatures and has useful creep–resistance. Depending on service conditions it can be used at temperatures up to 800°C.

The combined corrosion– and heat–resistance of Ni–Resist make it suitable for many high-temperature applications in the chemical, mining and processing industries.

LOW TEMPERATURE PROPERTIES

Ni–Resist retains to a marked degree its toughness and other mechanical properties at sub–zero temperatures. But, to avoid any danger of breakdown of the austenitic structure at very low temperatures, it is important that the nickel content should be not less than 14·5 per cent. in the standard grade, or 22 per cent. in the copper–free grade.

RESISTANCE TO WEAR AND GALLING

Ni–Resist is highly resistant to wear and galling in metal to metal contact. In this respect it is especially useful in heat and corrosive conditions. It also has marked resistance to erosion—as in pumps handling sandy sea water.

PRODUCTION

Ni–Resist can be produced in all the furnaces ordinarily used in the foundry for melting iron and steel. It may also often be produced economically in efficient non–ferrous foundry melting furnaces.

DESIGN

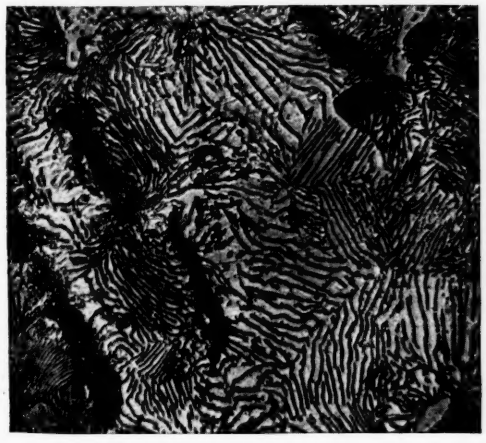
To minimize pattern and production costs, designs should be discussed with the founders. Abrupt changes in section should be avoided.

MACHINING

Ni–Resist tends to work–harden. Tools should be kept sharpened. Heavy, slow cuts are preferable. The finish is like that of a fine–grained grey cast iron. Special quality wheels are made for grinding Ni–Resist.

WELDING

Ni–Resist may be readily welded by the electric–arc or oxy–acetylene process using filler rods of nickel, Monel or Ni–Resist, Nilo 36 or Ni–Rod 55, as appropriate. Such welds are soft and machinable; welding rods of ordinary cast iron or steel must not be used.



× 500

(*a*) **Unalloyed iron showing pearlite with graphite flakes**

× 500

(*b*) **Fully austenitic cast iron showing graphite flakes in homogeneous corrosion-resistant matrix**

Table I

Composition and Properties of Ni-Resist Austenitic Cast Irons

	Standard	Copper–Free
Composition *per cent.*		
*Carbon	2·6–3·2	
Silicon	1·0–2·5	
Manganese	8–1·5	
Sulphur	0·12 max.	
†Phosphorus	0·40 max.	
Nickel	13–16	20–22
Chromium	1·5–3·0	
Copper	6–8	—
Physical Properties		
Specific Gravity	7·3	
Weight, *lb. per cu. in.*	0·264	
Mean Coefficient of Thermal Expansion, *millionths per °C.* 20°–200°C.	18·5	
Magnetic Response	Non-magnetic	
Thermal Conductivity, *c.g.s. units*	0·095	
Electrical Resistance, *microhms/cm./sq. cm.*	150	
Temperature Coefficient of Electrical Resistivity— *per °F.*	0·0001769	
per °C.	0·000318	
Pattern Shrinkage, *in./ft.*	$\frac{3}{16}$	
Mechanical Properties		
Tensile Strength, *tons/sq. in.*	10–14	
Trans. Strength 1·2 in. bar 18 in. span, *tons/sq. in.*	25	
Trans. Deflection 1·2 in. bar 18 in. span, *inch*	0·3–0·6	
Compressive Strength, *tons/sq. in.*	45–60	
Hardness (*B.H.N.*)	120–180	
Izod Impact, 1·2 in. bar struck 3 in. above support, *ft.–lb.*	Over 120	
Modulus of Elasticity, *millions of lb./sq. in.*	12–15	

NOTES ON COMPOSITION

Lead, tin and zinc are deleterious even when present in very small amounts. Care must therefore be taken to ensure their absence.

* Carbon content should be limited to a maximum of 2·9 per cent. where high strength and maximum density are essential.

† For certain applications, e.g., to give especially high resistance to wear in cylinder liners, phosphorus may be raised to 1·0 per cent. or higher, but at the expense of strength and toughness.

Table II

Ni-Resist Austenitic Cast Iron
Corrosion Test Results compared with Phosphor Bronze and ordinary Cast Iron

Figures represent the loss in milligrammes per square decimetre per day under static conditions at 20°C.

Corrosive Medium	Ni–Resist	Cast Iron	Phosphor Bronze	Relative Resistance to Attack	
				Ni–Resist to Cast Iron	Ni–Resist to Phosphor Bronze
Acetic Acid, 33%	17·0	840	18·6	49	1·1
Boracic Acid, 10%	7·7	57·4	4·6	7·5	0·6
Citric Acid, 5%	9·3	1,492	4·6	160	0·5
Formic Acid	13·9	138	13·9	10	1·0
Hydrochloric Acid, 1% ..	32·5	1,007	41·8	31	1·3
Hydrochloric Acid, 5% ..	54·2	3,360	57·3	62	1·1
Hydrochloric Acid, 20% ..	62·0	11,180	60·5	180	1·0
Nitric Acid, 1%	620	697	2,446	1·1	4·0
Nitric Acid, 5%	4,060	4,680	12,420	1·2	3·1
Nitric Acid, 20%	7,830	10,092	Dissolved	1·3	—
Oxalic Acid, 5%	6·2	55·8	12·4	9	2·0
Phosphoric Acid, 50% ..	26·4	4,650	7·7	176	0·3
Sulphuric Acid, 1%	26·4	1,642	18·6	62	0·7
Sulphuric Acid, 5%	37·2	6,880	37·2	185	1·0
Sulphuric Acid, 20%	41·8	13,720	38·8	328	0·9
Sulphurous Acid	240	1,032	9·3	4·3	0·04
Tartaric Acid, 5%	10·8	1,040	10·8	96	1·0
Vinegar	4·6	104	4·6	23	1·0
Acetone	1·5	4·6	1·5	3·1	1·0
Aluminium Sulphate, 5% ..	20·0	96·0	10·8	4·8	0·5
Ammonium Chloride, 5% ..	10·8	35·6	57·4	3·3	5·3
Ammonium Nitrate, 5% ..	51·2	163	57·4	3·2	1·1
Ammonium Sulphate, 10% ..	9·3	32·6	13·9	3·5	1·5
Ammonium Sulphate + 5% Sulphuric Acid	26·3	11,160	21·7	424	0·8
Carbon Tetrachloride ..	1·5	3·0	3·0	2·0	2·0
Copper Chloride, 10% ..	1,394	8,030	543	5·8	0·4
Ferric Chloride, 5%	667	1,038	347	1·6	0·5
Fuel Oil	1·5	1·5	1·5	1·0	1·0
Hydrogen Peroxide, 20 vols.	6·2	9·3	1·5	1·5	0·2
Magnesium Chloride, 10% ..	7·7	18·6	6·2	2·4	0·8
Magnesium Sulphate, 10% ..	3·1	14·0	3·1	4·5	1·0
Potassium Alum, 10% ..	15·5	372	20·2	24	1·3
Sea Water	6·2	23·2	6·2	3·7	1·0
Sodium Chloride, 3% ..	7·7	12·4	3·1	1·6	0·4
Sodium Hypochlorite ..	223	688	80·6	3·1	0·4
Sodium Sulphate, 5% ..	9·3	7·7	9·3	0·8	1·0
Sodium Sulphite, 10% ..	3·1	6·2	1·5	2·0	0·5
Sodium Sulphite, 5% ..	1·5	1·5	17·0	1·0	11

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Metallurgical Analysis: Textbook

W. A. NAISH, J. E. CLENNELL and V. S. KINGSWOOD: 'Select Methods of Metallurgical Analysis.' *Second (revised) edn., published by Chapman and Hall, Ltd.*, London, 1953; 660 pp. Price 75s.

Since the publication, in 1929, of the first edition of this textbook, there has been considerable alteration in approach to technical methods of analysis, and in revising the work the senior author enlisted the services of a panel of specialists in order to do full justice to all aspects of the subject. The revised edition is thus a combination of the set-up of the original book, amplified by material covering recent and specialized developments. Sectional contributions have been made in accordance with the following subdivisions:—analysis of iron, steel, and ferro-alloys; of aluminium and magnesium-base alloys; of copper-base alloys; of refractory materials; of coal and coke; determination of impurities in metals; analysis of iron ores and iron slags; physical methods of analysis and spectrographic methods; micro-analysis; electrolytic, electrometric, absorptiometric, polarographic, colorimetric and spectrophotometric methods.

While the aim of the book is to present a comprehensive account of every branch of metallurgical analysis, the authors have exercised critical selection, in order to include only methods which they and their collaborators consider to be consistently reliable. In addition to the detailed and well illustrated information given in the text, each chapter is supported by a *bibliography* of authoritative literature.

The section on analysis of nickel (pp. 317-22) includes procedures for determination of nickel, cobalt, silicon, iron, manganese, copper, sulphur and carbon; the section on analysis of elements includes (pp. 177-81) methods for gravimetric and volumetric determination of nickel. Methods for determination of nickel in copper, copper- and aluminium-base alloys, steel, tin, and white metals are given in the appropriate chapters.

Spectrochemical Analysis of Nickel-containing Materials

AMER. SOC. TESTING MATERIALS: 'Methods for Emission Spectrochemical Analysis.' *Published by the Society*, Philadelphia, Pa., Oct., 1953; 309 pp.

This publication, issued under the sponsorship of A.S.T.M. Committee E-2 (which is engaged on a programme of development aimed at standardization of spectrochemical practices and methods) presents, on a tentative basis, information and recommendations which have already been assembled in connexion with the Committee's work, and are considered to be potentially useful to laboratories, pending more complete standardization. Some of the procedures described are 'suggested methods' put forward by individuals or groups who have used them; other techniques have reached the stage of being proposed by Committee E-2 as 'tentative recommended methods'. The subject matter is classified in sections relating to (1) general practices in emission spectrochemical analysis, (2) proposals for standardization of nomenclature in this field, (3) procedures for spectrochemical analysis of metals and alloys, and of non-metals. Items of particular interest in relation to nickel-containing materials are as shown below—

Suggested Methods for Spectrochemical Analysis of:—
- Nickel Alloys by the Dry Powder–D.C. Arc Technique.
- Wrought Copper Alloys (including nickel-containing types) by the D.C. Arc Technique.
- Nickel Sheet by (*a*) the Point-to-Plane Spark Technique; (*b*) the A.C. Arc Technique.
- 70–30 Cupro–Nickel by the Cast Pin–Spark Technique.
- Low–Alloy Steels (including nickel-containing types) by various techniques.
- Alloy Cast Iron by the Cast Pin–Spark Technique.
- Nickel–Plating Solutions: Determination of Zinc by the A.C. Arc Technique.

Friction Sawing of Nickel-containing Materials

H. J. CHAMBERLAND: 'Friction Sawing Cuts Off High Costs.' *Iron Age*, 1954, vol. 173, Feb. 18, pp. 152-4.

This article urges the economic advantages to be gained by the use of friction sawing by the band method, drawing particular attention to improvements which have recently been achieved by introduction of a blade especially designed for this operation.

A table shows friction-cutting rates and saw-control factors recommended for cutting various materials, including nickel steels, nickel-chromium steels, and Illium (complex high-nickel corrosion-resisting alloy). Four typical sawing case histories are detailed to show the results obtained in (*a*) conventional sawing, and (*b*) friction sawing, of tool steel, 'special 90 per cent. nickel alloy', Monel, and chromium-vanadium steel strip.

Test for Susceptibility to Hot Cracking in Welding

See abstract on p. 71.

NICKEL

Extraction of Nickel: Review of Current Methods

A. R. BAILEY: 'Nickel Production.' *Metal Industry*, 1954, vol. 84, Jan. 8, pp. 23–4.

(One of a series on practice and recent developments in refining processes.)

This article gives a succinct account of the main features of the processes used by International Nickel Company, Mond Nickel Company, Ltd., Falconbridge Nickel Mines, Ltd., and Sherritt Gordon Mines Ltd., for extraction of nickel from sulphide ores. Reference is made to improvements recently introduced in the respective processes. Some analyses of commercial nickels on the market at various dates are given.

Extraction of Nickel viâ the Carbonyl

R. S. YOUNG: 'Purification of Nickel with Carbonyl: A Demonstration.' *Jnl. Chemical Education*, 1954, vol. 31, p. 26.

The writer describes technique and apparatus for lecture demonstration of gas-solid reactions involved in oxide reduction, low-temperature volatilization of metal, and dissociation of the gas formed, into metal and another gas. The reactions described are exemplified by reference to extraction of nickel by the carbonyl process, from nickel oxide.

Nickel Carbonyl and Related Compounds: Formation and Characteristics

'Metal Carbonyls.'

R. NAST and T. VON KRAKKAV: 'LVI. Formation of Nickel Tetracarbonyl from Potassium Hexacyanodinickelate.'

R. NAST and H. ROOS: 'LVII. Nickel Carbonylcyano Complexes.'

Zeitsch. f. anorganische Chemie, 1953, vol. 272, pp. 233–41, 242–52.

Summary of this work is given in *Chemical Abstracts*, 1953, vol. 47, Nov. 25, pp. 12,090–1.

Stereochemistry and Valency States of Nickel

R. S. NYHOLM: 'Stereochemistry and Valency States of Nickel.' *Chemical Reviews*, 1953, vol. 53, pp. 263–308.

A comprehensive review, supported by a *bibliography* of 212 references.

Magnetostriction in Nickel and Nickel-Copper Alloys

See abstract on p. 67.

Cobalt in Raney Nickel Catalysts

J. SFIRAS and A. DEMEILLIERS: 'Study of the Influence of Cobalt on the Selectivity of Nickel Raney Catalysts.' *Recherches*, 1953, Oct., p. 32.

Following observations of unexplained selectivity in the effect of nickel catalysts in certain reactions, the authors of this note explored the possibility that the presence of cobalt in the nickel might be the explanation. Three alloys were prepared, containing, respectively:—

Nickel %	Cobalt %	Aluminium %
50	—	50
48	2	50
45	5	50

From these alloys Raney-type catalysts were made by standard methods. Their behaviour was studied as affecting the partial hydrogenation of citral under low pressure. Practically identical results were obtained with all three catalysts, indicating that the presence of the proportions of cobalt used had no selective effect.

Influence of Hydrogen Sulphide on Activity of Nickel Catalysts

H. A. DIRKSEN, H. R. LINDEN and E. S. PETTYJOHN: 'Cracking Catalyst Activity in the Presence of Hydrogen Sulphide.' *Inst. Gas Technology, Research Bull. No. 4*, 1953; 27 pp.

In the search for a catalyst which should be highly active, mechanically stable, and sulphur-resistant in the steam-hydrocarbon reaction, oxides and sulphides of nickel, iron, cobalt, chromium, molybdenum, tungsten, vanadium, niobium and tantalum were prepared, with thorium oxide and zirconium oxide as promoters. Al_2O_3, Al_2O_3-SiO_2, MgO, Filtrol, MgO-Al_2O_3, SiO_2, SiO_2-MgO, and MgO-infusorial earth were used as supports. Details of mode of preparation of the catalysts, experimental conditions, and conclusions drawn from the tests are summarized in *Chemical Abstracts*, 1953, vol. 47, Nov. 10, pp. 11,700–1.

Nickel in its various forms was found to be the most promising catalyst for the steam-propane reaction, followed by molybdenum. Iron, chromium and cobalt were generally less active. In the presence of hydrogen sulphide the sulphide forms of the catalysts were normally more active than the corresponding oxide forms.

Influence of Composition of Nickel Core on Behaviour of Oxide Cathodes

G. H. METSON and M. F. HOLMES: 'Deterioration of Valve Performance due to Growth of Interface Resistance.' *Post Office Electrical Engineers' Jnl.*, 1954, vol. 46, Jan., pp. 198–9.

The growth of a resistive interface layer between an oxide cathode and its supporting core is generally accepted as being one of the primary causes of deterioration of performance of high-slope receiving valves. The authors of this note discuss the magnitude of the effect occurring in practical operation, and give an account of experiments in which interface resistance was eliminated by replacement of the nickel core normally used by one of platinum free from silicon. A typical core material used in receiving valves is nickel

containing 0·05 per cent. of magnesium and 0·05 per cent. of silicon. During processing the silicon content of the core metal reacts with the oxide matrix, to form the interface layer of barium orthosilicate. At the beginning of life this orthosilicate layer is activated by excess barium atoms, and it is the progressive deactivation of this semi-conducting layer, during life, which leads to the observed rise of interface resistance. The deactivation may occur either by diffusion of excess barium out of the layer, or by its destruction *in situ* by an invading oxidizing gas. Experiments carried out by the authors lead them to the conclusion that the second cause is the operating factor in working valves. Tests up to 17,000 hours on valves having a core of silicon-free commercial-grade platinum showed no measurable increase in interface resistance, and in view of such confirmation that silicon is the harmful constituent, efforts are now being directed towards production of nickel alloys to the following specification: magnesium 0·05–0·15, silicon less than 0·001, per cent., with other impurities not exceeding the limits found in typical core materials such as 'O' nickel.* Although the setting of this low upper limit to the silicon content presents some difficulties in manufacture, it is considered to be desirable, since, at a conservative estimate, it appears likely that elimination of growth of interface resistance may double the life of the common high-slope receiving valve.

Nickel for Electronic Valves

'Nickel for Electronic Valves.' *Engineer*, 1954, vol. 197, Jan. 22, pp. 151-2.

Two new grades of nickel suitable for the manufacture of electrodes and other parts of valves have recently been placed on the market by Messrs. Henry Wiggin and Company, Ltd. The compositions are shown below.

	H.P.A. Nickel %	H.P.B. Nickel %
Carbon	0·10 max.	0·10 max.
Copper	0·04 ,,	0·04 ,,
Iron	0·05 ,,	0·05-1·0
Manganese ..	0·02 ,,	0·10 max.
Magnesium ..	0·01 ,,	0·01 ,,
Silicon	0·03 ,,	0·15-0·25
Aluminium ..	0·01-0·05	0·02 max.
Sulphur	0·005 max.	0·005 max.
Nickel+Cobalt ..	99·5 min.	99·5 min.

The H.P.A. grade, available in the form of strip, tape and wire, is specially recommended for components of valves operating at high temperatures in applications in which evaporation of volatile elements such as magnesium cannot be tolerated. It is also useful for cathodes of valves which are required to have very long life, while delivering only low emission current.

H.P.B. nickel is intended primarily for cathodes where sublimation of magnesium cannot be tolerated on account of very small inter-electrode distances, or where the operating temperature of the cathode is higher than usual and leads to excessive evaporation of magnesium. The high silicon content of this grade of nickel ensures ready activation and good emission, but the warning is given that care must be exercised in the running of valves having cathodes of H.P.B. nickel, since, under certain conditions, the high silicon content can result in the build-up of high interface impedances between the cathode sleeve and the coating material.

* Nickel+cobalt 99·5 min., copper 0·1 max., iron 0·2 max., manganese 0·15 max., carbon 0·04 max., sulphur 0·005 max., per cent.

Nickel Waveguides

F. A. BENSON: 'Attenuation in Nickel and Mild-Steel Waveguides at 9 375 Mc/s.'
Proc. Instn. Electrical Engineers, Part III, 1954, vol. 101, Jan., pp. 38–41.

The author reports measurements of the attenuations produced by air-filled waveguides of nickel and mild steel, at a frequency of 9 375 Mc/s. It is shown that the permeability of a ferromagnetic material can be determined from such measurements, provided that the roughness of the internal surfaces of the waveguide can be estimated with reasonable accuracy. It is found that mild-steel waveguides having machined internal surfaces give attenuations as high as 3.79 db/m, and the figure of 3 is deduced for the permeability of commercially-pure nickel drawn waveguides.

Determination of Traces of Magnesium in Nickel

C. L. LUKE and M. E. CAMPBELL: 'Determination of Magnesium in Nickel.' *Analytical Chemistry*, 1954, vol. 26, Feb., p. 424.

Abstract of paper presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March, 1954.

'There exists an urgent need for a good photometric method for determination of traces of magnesium in nickel for electronic applications. The authors present details of procedure developed to meet this need. It consists in solution of the sample in nitric acid, removal of all interfering ions by classical separations, and determination of magnesium by extraction with an oxine-chloroform solution from ammoniacal solution, and photometric measurement of the yellow colour of the extracted magnesium compound.'

Spectrochemical Analysis of Nickel-containing Materials

See abstract on p. 62.

Metallurgical Analysis: Textbook

See abstract on p. 62.

ELECTRODEPOSITION AND OTHER COATING METHODS

Determination of Nickel in Plating Solutions

K. E. LANGFORD: 'The Determination of Nickel in Nickel Plating Solutions using Disodium Ethylenediamine Tetra-Acetate.' *Electroplating*, 1954, vol. 7, Feb., pp. 46–8.

The titration of certain metal ions, particularly calcium and magnesium, using E.D.T.A., was described in 1950, by HEALD *et al.*, and methods for analysis of plating solutions based on such techniques have been developed by the present author (*ibid.*, 1952, vol. 5, p. 41 and 1953, vol. 6, p. 41). The applicability of the method to metal ions other than calcium and magnesium depends on the discovery and exploitation of suitable indicators sensitive to the presence of the particular ion being titrated. To date only two such indicators are widely employed (1) Eriochrome Black T, and (2) Murexide (ammonium purpureate), sensitive, respectively, to magnesium and to calcium ions. It has been shown that murexide is sensitive also to nickel ions, indicating that under correct conditions it should be possible to titrate nickel with E.D.T.A. This paper reports experiments made to determine the optimum conditions for such analysis. The method developed, of which full working details are given, is applicable to both dull- and bright-type nickel-plating solutions, and by introduction of suitable modifications the titration can be performed in the presence of cobalt. Magnesium interferes, and although by an alternative procedure moderately accurate results can be obtained, the method is not recommended in the presence of this cation.

H. FLASCHKA: 'E.D.T.A. in Metallurgical Analysis, especially for the Determination of Nickel and Zinc.' *Chemist Analyst*, 1953, vol. 42, Dec., pp. 84–6.

The author briefly refers to the growing importance of ethylenediamine tetraacetic acid (E.D.T.A.) in analysis, and to some of the relevant literature.

E.D.T.A. is a relatively non-specific agent, capable of complexing most di- and tri-valent ions. It is therefore necessary to make separations, or to use other complexing agents to mask some of the other metals present in a solution which is being analysed for a specific element. In many cases separations are inevitable and in such cases a simple and rapid method for titration of the separated metal must be sought. HARRIS and SWEET (*Analytical Chemistry*, 1952, vol. 24, pp. 1062–3: *Nickel Bulletin*, 1952, vol. 25, Nos. 8–9, p. 213) determined nickel in steel by precipitating in the usual manner with dimethylglyoxime and then treating the organic nickel salt with hydrochloric acid and evaporating to dryness, to destroy the organic component. This procedure requires a relatively long time, and the present author suggests a more rapid method, based on the fact that nickel dimethylglyoximate is not precipitated from an ammoniacal solution containing E.D.T.A. Excess of E.D.T.A. can be back titrated with a standard zinc-sulphate solution, using Eriochrome Black T as indicator. Details are given below:—

'*Reagents*

'*E.D.T.A. Solution, 0·1M.* Dissolve 37·22 g. of E.D.T.A. disodium salt dihydrate in distilled water and dilute to one litre.

'*Zinc Solution, 0·1M.* Dissolve 28·755 g. of $ZnSO_4.7H_2O$ in distilled water, and dilute to one litre.

'*Buffer Solution.* Dissolve 13·4 g. of ammonium chloride and 88 ml. of aqueous ammonia (d. 0·91) in water, and dilute to 250 ml.

'*Indicator Powder.* Grind one part of Eriochrome Black T with 400 parts of sodium chloride, to a fine powder.'

'*Procedure for Determination of Nickel*

'Precipitate nickel in the usual manner with dimethylglyoxime, filter, and wash. Dissolve the precipitate on the filter with hot, dilute hydrochloric acid and rinse the filter repeatedly with water. Add to the acidic filtrate an excess of 0·1*M* E.D.T.A. solution, neutralize with aqueous ammonia, using a little methyl red, and add 5–10 ml. of buffer solution for each 50–80 ml. of solution. Add sufficient indicator powder to produce an easily visible blue colour, and titrate the excess of E.D.T.A. with zinc solution until the colour changes from blue to red. (If too much methyl red has been taken for the neutralization this indicator causes a yellow background and the colour changes to blue-green or even green, without any effect on the accuracy of the results.)

'If *A* is the ml. of 0·1*M* E.D.T.A. solution, *B* the ml. of 0·1*M* zinc solution, and *W* the weight of the sample in mg, the per cent. nickel is given by the expression $586{\cdot}9\ (A—B) \div W$.

'*Alternative Procedure for Nickel*

'In order to avoid the need for two standard solutions, add to the acid solution of the nickel dimethylglyoximate the potassium magnesium salt of E.D.T.A. (MgK_2Y), and after buffering titrate the magnesium liberated with E.D.T.A. and Eriochrome Black T. This method is especially recommended for micro-determinations.'

Typical analytical results are given in support of the procedure described, and recommendations are made for determination of zinc.

Determination of Boric Acid in Nickel-Plating Baths

G. GABRIELSON: 'The Determination of Boric Acid in Nickel Plating and Acid Zinc Plating Baths by Means of Cation Exchangers.' *Plating*, 1954, vol. 41, Jan., pp. 47–8, 53–4.

SERFASS *et al.* (*ibid.*, 1953, vol. 40, p. 59) have shown that nickel interferes with determination of boric acid in plating solutions, and have indicated that there are three ways of removing nickel: (1) deposition by electrolysis, (2) precipitation, and (3) formation of complex nickel compounds. They recommend precipitation of nickel with potassium ferrocyanide, followed by titration with standard alkali, without filtration of the solution. The present author suggests an alternative method for removal of nickel ions, by

percolating the solutions through a layer of a hydrogen-saturated cation exchanger, so that all cations present are exchanged for hydrogen ions. The theory and practical application of the method are covered in this paper.

Spectrochemical Determination of Nickel in Plating Solutions

See abstract on p. 62.

Mechanical Properties and Corrosion-Resistance of Plated Aluminium-base Alloys

H. PAIGE, J. H. JAMES and F. S. WILLIAMS: 'How Tough are Nickel and Chromium Electroplates for Aluminum?' *Product Engineering*, 1953, vol. 24, Dec., pp. 162–7.

As a means of widening the scope of application of light alloys, electroplating is attracting considerable attention, and among the most promising coatings for aluminium-base materials are nickel and nickel + chromium. The tests reported in this paper, carried out in the U.S. Naval Air Experimental Station, Philadelphia, were made to ascertain the influence, on mechanical properties and resistance to corrosion, of nickel coatings deposited, to various thicknesses, from four different types of bath, and of nickel + chromium coatings, all such coatings being deposited on a zincated copper-flashed aluminium-alloy surface. Chromium coatings deposited over copper without a nickel undercoat, or direct on a zincated aluminium surface were also vested.

The results of *mechanical and fatigue tests* are given in a series of graphs and tables showing the behaviour of specimens as plated and after exposure to salt-spray and atmospheric corrosion attack. Some of the major conclusions drawn from the results are summarized below:—

In general, no substantial changes in ultimate tensile strength and yield strength were found in the unexposed plated specimens with coatings up to about 8 mils in thickness. With heavier deposits, however, strength usually fell with increasing thickness of plating. All deposits, with the exception of the nickel + chromium coating, caused reduction in ductility and percentage elongation, even at low thicknesses: elongation of the nickel + chromium-plated specimens was superior to that of the specimens carrying a coating of nickel only. Chromium coatings applied directly on the zincated surface of the aluminium alloys shattered and completely flaked off, in marked contrast to the strong adherence shown by chromium-plated specimens on which a copper interlay had been used between the zinc film and the chromium. Strong adhesion was also observed in the copper + nickel + chromium-plated specimens. Percentage elongation was markedly lowered in the specimens tested after corrosion exposure, but again the nickel + chromium-plated samples showed the best behaviour. The fatigue tests showed that the strength depends on the type of coating as well as on its thickness: specimens carrying bright nickel coatings exhibited definite superiority over other types. The reason for this difference is discussed in relation to the nature of the stresses (internal and applied) acting on the specimens. In specimens on which duplex nickel + chromium coatings had been deposited a bright-nickel underlay did not have the same favourable influence on fatigue-resistance, indicating that behaviour is determined rather by the final than by the under coating.

Corrosion Tests. In salt-spray and atmospheric corrosion tests, specimens carrying a coating of 2 mils nickel, 2 mils chromium displayed the most outstanding resistance. On the basis of a given thickness of plating, nickel coatings gave the next best protection, e.g., heavy layers of nickel (8 mils) remained unaffected after 4,000 hours' exposure to salt spray. In salt spray thinner deposits of nickel suffered pitting attack in shorter periods, but nickel coatings only 1 mil thick gave some protection in atmospheric exposures of 16 months' duration. Chromium deposited over a copper strike undercoat, without nickel, was markedly less resistant to corrosion than nickel. After 2,000 hours' exposure to salt spray marked pitting had developed in coatings up to 4 mils thick, and after 4,000 hours even the specimens carrying a coating of 8 mils contained some pits. Atmospheric exposure of the copper + chromium-coated specimens caused severe corrosion in the underlying aluminium, resulting in formation of voluminous deposits which eventually produced buckling of the chromium layer. In the salt-spray tests chromium deposits without copper undercoat behaved in a manner similar to that found in those on which a copper underlay had been used: in weathering tests the copper-free specimens were superior, but, as noted above, the adherence was poor. Chromium coatings deposited direct on aluminium prepared only by mechanical roughening of the basis surface showed severe corrosion after 1000 hours of salt-spray exposure and the plating broke off, due to swelling of the corrosion product formed under it. The net conclusion from the series is that nickel + chromium deposits of adequate thickness have outstanding merit as a means of protecting aluminium-base materials against corrosion.

Porosity Testing of Electrodeposited Nickel Coatings

F. OGBURN and A. BENDERLY: 'The Nature, Cause and Effect of the Porosity in Electrodeposits. 1. The Porosity of Electrodeposits.' *Plating*, 1954, vol. 41, Jan. and Feb., pp. 61–5, 169–73.

This report is issued under the aegis of A.E.S. Research Project 13, which was initiated to study the applicability of the gas-permeability method as a means of determining the protective value of electrodeposited coatings. The investigation of initial permeability was limited to thin coatings, since thick deposits were known to be virtually impermeable to gases.

The authors describe the gas-permeability test apparatus used and present typical data obtained by this method. They also give details of a photographic method, developed initially as an adjunct to the gas-permeability procedure, but found to be, in some respects, superior to it. The sensitivity and reliability of the two methods are compared on typical nickel foils comprising electrodeposited coatings detached from the basis metal.

For reasons which are deduced from the results of these measurements and other known characteristics of electrodeposited coatings, the authors conclude that evaluation of the protective value of a coating by measuring the initial porosity of a detached foil (by either gas-permeability or the photographic method) has serious limitations, and that the results would be unlikely to give an accurate forecast of service behaviour. They therefore instituted tests in which comparison was made of the rates at which foils become porous when corroded. In the first three series hydrochloric-acid vapours were employed, under varying conditions, as the corrodent: these experiments were supplemented by hot-water immersion corrosion tests and by tests in which the specimens were immersed in sulphuric-acid solution, 10 per cent. by volume.

From this group of tests it was concluded that (1) selection of a suitable accelerating corrodent which will simulate atmospheric corrosion or a particular type of service condition may be difficult, and (2) since the pinhole type of corrosion occurs non-uniformly, with unattacked areas between the pores, the use of a method like the gas-permeability procedure, which tests only a small area, may lead to erroneous conclusions.

Observations on outdoor corrosion of adherent nickel deposits and detached foils have indicated that pinholing or depth corrosion is considerably more prominent than surface corrosion. It is therefore desirable that accelerated tests should be such as to develop a similar relationship between depth and surface corrosion. Immersion tests in hydrochloric-acid solution, employed by some investigators, have been found by the present authors to be somewhat unsatisfactory, since the deposit thickness is reduced 25-50 per cent. before measurable porosity is developed by hydrochloric acid. In the present investigation several other corrodents were used, in comparison with 1*N* hydrochloric acid, viz., 2*N* sulphuric acid, 0·5*N* sodium chloride, and 2*N* acetic acid. The conclusions derived from these tests are summarized as follows:—

(*a*) Hydrochloric acid causes the most rapid rate of surface corrosion (as determined by loss in weight), but rather slight depth corrosion (determined by photographs).
(*b*) Acetic acid results in slight surface corrosion, but extreme depth corrosion.
(*c*) Sulphuric acid is intermediate between (*a*) and (*b*).
(*d*) Salt solution results in extremely little depth or surface corrosion.

The behaviour of the foils in the acetic-acid solution led to the conclusion that the conductivity of the corrodent was related to its tendency to produce depth-type corrosion, and later experiments, with acetic, formic, and butyric acids, confirmed this conclusion. As conductivity of the solution was increased the extent of depth corrosion of the foil decreased, but increase in conductivity resulted in greater weight loss, indicating that depth corrosion only was inhibited by such modification, whereas total corrosion actually increased. The authors consider that the relation between conductivity of the corrodent and the nature of the resultant corrosion merit further study.

Finally, they draw attention to the difficulty in correlating the conclusions drawn from work on detached electrodeposited foils with the behaviour of adherent deposits employed under service conditions. Work is in hand in an attempt to detect the porosity of intact adherent coatings by radioactive methods, the proposal being that a thin radioactive coating should be deposited on the basis metal and that a regular coating should then be superimposed. Autoradiographs would be made, on the presumption that pores in the upper coating would appear on film as spots similar to those now resulting from conventional photographs made of detachable coatings. Although such a method could not be expected to find general application, it is considered that it might prove useful as a research tool.

NON-FERROUS ALLOYS

Magnetostriction in Nickel and Nickel-Copper Alloys

K. AZUMI and J. E. GOLDMAN: 'Volume Magnetostriction in Nickel and its Alloys.' *Bull. Amer. Physical Soc.*, 1954, vol. 29, Jan., p. 44.

Abstract of paper presented at 1954 meeting of the American Physical Society. Measurement of volume magnetostriction in nickel and two nickel-copper alloys.

Magnetic Susceptibility of Nickel-Copper and Nickel-Chromium Alloys

J. E. GOLDMAN and A. ARROTT: 'Magnetic Susceptibility of Nickel-Copper and Nickel-Chromium Alloys.' *Bull. Amer. Physical Soc.*, 1954, vol. 29, Jan., p. 45. Abstract of paper presented at 1954 meeting of the American Physical Society.

'Extrapolation of the magnetic properties of nickel-copper and nickel-chromium alloys measured at high concentrations of nickel suggests that the spontaneous magnetization observed at 0°K. (−273°C.) should disappear at 60 per cent. of copper and about 14 per cent. of chromium, respectively. The nature of the magnetism in the alloys having higher concentrations of the non-ferromagnetic constituent is not clearly understood. The authors made measurements of magnetization down to 2°K. (−271°C.) on specimens of alloys in that range of composition. They found the nickel-copper alloys containing 58 and 63 at. per cent. of copper to be ferromagnetic: the temperature-dependence of this ferromagnetism was studied. In the case of nickel-chromium alloys the behaviour is paramagnetic, and possibility of ferromagnetism or antiferromagnetism seems to be precluded. The results are discussed on the basis of the collective electron treatment, the parasitic paramagnetism of Néel, and the rôle of possible concentration fluctuations within the material.'

Permanent-Magnet Materials: Reference Data

See abstract on p. 68.

Nickel-Gallium Alloys

W. B. PEARSON: 'A Nickel-Gallium Superlattice (Ni_3Ga).' *Nature*, 1954, vol. 173, Feb. 20, p. 364.

The nickel-gallium system was studied in 1950 by HELLNER, who stated that the nickel solid solution extended to 28 at.-per cent. gallium, while the lattice parameter increased to 3·591Å at the solubility limit. The writer of the present note has found a Ni_3Ga phase, having the Cu_3Au superlattice structure. The nickel-gallium system is similar to the nickel-aluminium system, in that the ordered phase has a relatively small homogeneous range, while on the nickel-rich side a wide heterogeneous range is found at low temperatures, producing a considerable difference of lattice parameter of the two co-existing cubic phases. Fuller details of the research will be published later.

Solid-State Bonding of Aluminium and Nickel

S. STORCHHEIM, J. L. ZAMBROW and H. H. HAUSNER: 'Solid-State Bonding of Aluminum to Nickel.' *Jnl. of Metals*, 1954, vol. 6, Feb., Sect. 2; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 269–74.

This is the full form of the paper to which reference was made, from an abstract source, in *Nickel Bulletin*, 1953, vol. 26, No. 10, p. 170.

It is concerned with the effect of processing variables (pressure, temperature, and time at pressure) on solid-state bonding. The method employed was that of hot-pressing, and the metals used were aluminium and nickel. Special attention was given to study of the strength of the bond between the two metals and to the intermetallic penetration rate. In view of its practical significance, the effect of pressure was studied in particular detail.

Two different types of specimen were made, one for subsequent tensile test, the other for investigation of intermetallic penetration. Details are given of preparation and of the hot-pressing conditions.

Results are recorded at length: some of the principal observations made and conclusions drawn are summarized below:—

Ultimate Tensile Strength

Curves show results on specimens pressed at 400°–600°C., at different applied pressures: 2, 11, and 20 tons per sq. in., each applied for 4 minutes. Increasing temperature of pressing resulted in increase in tensile strength (specifically in specimens pressed at 11 and 21 tons per sq. in.). The curves reach a maximum for a temperature of about 500°C., subsequently fall, and then show a levelling-off period for pressing temperatures above about 550°C. For a given temperature of pressing, tensile strength rises with increase in applied pressure, and beginning of incipient bonding occurs at lower temperatures as the pressure is increased: under 11 and 20 tons per sq. in. it occurred at 400°C. At 450°C. tensile strength rose rapidly with increase in applied pressure, and at 500°C. the bonding increased even more rapidly as a function of pressure, and rose to much higher values. At 550°C. the increase in strength as related to pressure was more gradual, and at 600°C. values obtained during 4-minute pressing were not so high as those produced by corresponding pressures at 500°C.

Examination of the effect of time (up to 12 minutes) at pressure (11 tons per sq. in.) showed a maximum with a holding time of 4 minutes, and the highest values were obtained with such conditions at 500°C. operating temperature.

Intermetallic Penetration

In the studies on intermetallic penetration, the most important finding was that, as in the case of the tensile properties, increased pressure has a marked influence on penetration rates. The results of metallographic examination showed that by the use of sufficiently high pressures it is possible to prevent formation of embrittling intermetallic phases, indicating a means of overcoming the difficulties previously encountered in bonding materials likely to give rise, on penetration, to brittle layers. This aspect is being followed up, as being of significance from the practical as well as the academic point of view, with the hope that more basic theoretical understanding of the phenomena involved will lead to useful advances in the technique of solid-state bonding.

Metallurgical Analysis: Textbook

See abstract on p. 62.

Spectrochemical Analysis of Nickel-containing Materials

See abstract on p. 62.

NICKEL-IRON ALLOYS

Permanent-Magnet Materials: Reference Data

D. HADFIELD: 'Permanent-Magnet Steels and Alloys.' *Metal Treatment and Drop Forging*, 1954, vol. 21, Jan., pp. 15–24.

Preliminary consideration is given to the essential characteristics of permanent-magnet materials and to the theory underlying the development of the various types currently used. A historical survey is then made of work in all parts of the world from which advances in this field have stemmed, with due reference to research in Japan, England, Holland, Germany, and U.S.A. Comprehensive tables show the compositions and properties of steels, alloys and other materials used for permanent magnets, with indication of the date of discovery and the country of origin. Production methods used for magnets of the respective types, and some of the treatments which are applied to secure directional and other properties, are discussed. The information given affords striking demonstration of the importance of the nickel-aluminium-cobalt-iron-base and other nickel-containing magnet alloys.

J. C. WILLIAMSON: 'Steels for Permanent Magnets. Survey of the Main Types.' *Electrical Rev.*, 1953, vol. 153, Nov. 13, pp. 1101–5.

In this review of the current stage of development of permanent magnets the author briefly describes the production and properties of magnets of the older types, made from quench-hardening chromium and cobalt steels, and gives more detailed attention to the

Composition and Principal Magnetic Properties of Permanent-Magnet Materials

Material	Approximate Composition %					Average Magnetic Properties		
	C %	Co %	Cr %	Mo %	W %	Brem	(BH)max	Hc
Chromium steel	0·9	—	3	—	—	9,800	285,000	70
15% Cobalt steel	1·0	15	9	1·5	—	8,200	620,000	180
35% Cobalt steel	0·9	35	5	—	4	9,000	950,000	250
	Ni %	Co %	Al %	Cu %	Nb %			
Alni	25	—	12	—	—	5,600	$1·25 \times 10^6$	580
Alnico	16	12	9	5	—	7,250	$1·7 \times 10^6$	560
Hycomax	21	20	9·5	2	—	9,000	$3·2 \times 10^6$	850
Alcomax II	11	24	8	5	—	12,400	$4·5 \times 10^6$	575
Alcomax III	13	24	8	3	1	12,500	$5·0 \times 10^6$	670

newer forms of magnet material, of the nickel-aluminium-iron-base types.

The latter alloys are usually melted in small furnaces, to facilitate the close control of composition which is essential to realization of optimum properties. A basic-lined furnace is preferred, because in such materials silicon is generally harmful, and very pure materials must be used for the charge, since even minor amounts of many elements can cause noticeable reduction in magnetic quality. The magnets are mostly made as individual castings, although thin bars are occasionally cut into short lengths, and in some cases the 'chocolate-bar' method of a grooved slab subsequently broken up is used, taking advantage of the inherent brittleness of these alloys. Heat-treatment consists in cooling from a high temperature, in order to produce a single-phase structure, followed by heating in the range 500°-700°C., to cause precipitation of a second phase. In the most recently developed magnets of this type greatly enhanced magnetic strength in one direction is secured by the application of a magnetic field during cooling: the improvement of properties so producible is illustrated in the table above. After heat-treatment the magnets are ground (the only form of machining which can be applied to these alloys). The exceptional qualities of the newer magnets make possible their use in various applications in which use of electromagnets was previously inevitable, and in locations in which the introduction of leads required for operation of electromagnets would be difficult. A further highly important characteristic is their stability. The cobalt steels and other types of material used earlier are in an unstable condition after quenching, and magnetic strength decreases during use, slowly at ordinary temperatures and much more rapidly as the temperature is raised. In the nickel-aluminium-iron alloys, however, no measurable change of structure takes place at normal temperatures, and the slight loss occurring on heating is reversed on cooling if the temperature to which the material has been subjected is not above about 500°C. These newer alloys are also much more stable against the influence of magnetic fields: this feature is discussed by the author on the basis of typical demagnetization curves for the older and newer magnet materials, and the relative importance of remanence and coercivity is also considered.

Nickel-Iron-base (Isoelastic) Alloys for Ultrasonic Delay Lines

'Ultrasonics: Thermally Stable Delay Line may be Possible.' *Iron Age*, 1954, vol. 173, Feb. 11, pp. 154–6, 158: see also full report in *Nat. Bur. Standards Jnl. of Research*, 1953, vol. 51, Nov., pp. 209-20; R.P. 2453.

The National Bureau of Standards has recently been investigating metal ultrasonic delay lines for use in electronic computers and ordnance devices, with particular reference to attenuation, distortion, and temperature-response. A primary essential is temperature stability over the range $-50°$ to $+200°$C.

Materials tested in this connexion include two magnesium alloys, nickel (high-purity and commercial grades), 32 per cent. nickel nickel-iron alloy, 18–8 chromium-nickel steel, Invar (36 per cent. nickel nickel-iron alloy), 1 per cent. carbon steel, aluminium single crystals, and various grades of Isoelastic alloy (iron-base alloys containing about 36 per cent. of nickel and 7–8 per cent. of chromium, with minor percentages of other, unspecified, constituents). Of all the materials examined, two of the Isoelastic alloys were the only ones showing the required degree of stability over the full temperature range specified.

Metallurgical Analysis: Textbook

See abstract on p. 62.

CONSTRUCTIONAL STEELS

High-Strength Low-Alloy Steels: American Types

'High-Strength, Low-Alloy Steels.'
Materials and Methods, 1954, vol. 39, Feb., pp. 117-32, (*Materials and Methods Manual* No. 102).

This manual covers the general characteristics, corrosion-resisting qualities, mechanical and physical properties, and working and joining characteristics of representative high-tensile low-alloy steels of types currently manufactured in U.S.A. under the following designations:—

Aldercor, Cor-Ten, Republic Double-Strength, Dynalloy, Hi-Steel, Mayari-R, N-A-X High-Tensile, Otiscoloy, Tri-Ten and Tri-Ten 'E', Yoloy.

The steels contain low percentages of the following elements, in alloying quantities, used in various combinations and proportions: manganese, copper, nickel, chromium, molybdenum, zirconium, phosphorus.

Considerations involved in designing with such steels are discussed, and a concise review is made of some of the major applications in which they have been successfully used, e.g., railway stock, automobile and agricultural equipment, structural engineering, ships, and general engineering. The economics of the subject are considered on the basis of first cost per unit of strength, cost per unit of service life, and saving in operating costs.

(It may be noted that although high-strength, low-alloy steels are discussed in this manual solely as exemplified by American types, many steels having similar characteristics are in production also in other countries.)

Metallurgical Analysis: Textbook

See abstract on p. 62.

Spectrochemical Analysis of Nickel-containing Steels

See abstract on p. 62.

Friction Sawing of Nickel-containing Steels

See abstract on p. 62.

HEAT- AND CORROSION-RESISTING MATERIALS

High-Temperature Alloys: Developments in U.S.A.

C. L. CLARK: 'American Developments in Alloys for High Temperatures.' *Metal Progress*, 1954, vol. 65, Jan., pp. 72-6.

A general review article, discussing types of material which are currently finding application in U.S.A. in high-temperature applications. The properties characteristic of the following materials, and their respective spheres of use, are considered:—lower-alloy steels (e.g., of the molybdenum, chromium-molybdenum, and chromium - molybdenum - vanadium grades); high-chromium steels containing small amounts of other elements; higher-alloy steels of the austenitic types, and the nickel- or cobalt-base alloys which have been developed mainly for the gas-turbine field. Nominal compositions and typical properties are given for representative materials in each category.

Critical consideration of the present stage of development indicates the need for (1) evolution of a chromium-nickel austenitic steel or alloy having still greater strength at high temperatures than those at present available for the steam-power and chemical-engineering industries, and (2) production of alloys capable of withstanding the ever-increasing temperatures to which gas-turbine components are being subjected, or, alternatively, the devising of cooling methods by which such components can be maintained, during service, at temperatures at which known materials can operate satisfactorily.

Heat-Resisting Nickel-Chromium-Iron Alloy

R. W. BORING: 'Low-Cost Alloys Offer Good Heat-Resistance.' *Iron Age*, 1954, vol. 173, Feb. 18, pp. 137-41.

This article urges the suitability of the 35-15 type of nickel-chromium-iron alloy as a heat-resisting material, exemplified in particular by an alloy designated R.A.330, which is stated to have the following composition:—carbon 0·20 max., manganese 2·00 max., silicon 0·75-1·50, chromium 14·5-16·5, nickel 34·0-37·0 per cent., balance iron, with or without small amounts of addition elements such as molybdenum, tungsten, titanium, niobium. The individual influence of each of the basic elements present in the alloy is reviewed. The material is primarily intended for use in the range 1500°-2150°F. (815°-1175°C.): it may be used at temperatures up to 1900°F. (1035°C.) in oxidizing atmospheres, and up to 2150°F. (1175°C.) in neutral or reducing atmospheres free from sulphur. Typical mechanical properties are quoted for R.A.330 at 1200°, 1400°, 1600° and 1800°F. (650°, 760°, 870° and 980°C.) and some recommendations are made with regard to design stresses at various temperatures.

Lattice-Parameter Measurements during Oxidation of Nickel-Chromium Alloys

W. C. HESELWOOD and K. W. ANDREWS: 'Lattice-Parameter Measurements and the Rate of Oxidation of Alloys.' *Bull. Inst. Metals*, 1954, vol. 2, Jan., pp. 65-6.

X-ray diffraction photographs have been used for identification of the oxides formed on the surface of metals and alloys at high temperatures, using the thickening of the scale and accompanying intensity of the diffraction lines due to the oxide phase and the decreasing intensity of the pattern of the underlying metal as qualitative measures of oxidation. In certain alloys a more quantitative measurement may be obtained by measurement of the lattice parameter of the underlying alloy, provided that oxidation is selective and that there is sufficient change in lattice parameter with composition. A typical case in which this method may be used is the nickel-chromium alloys, since in these materials there is a clearly marked

change in lattice parameter as oxidation proceeds. The authors of the note show graphs illustrating the changes occurring in seven different qualities of 80-20 nickel-chromium electrical-resistance alloy, subjected to the standard Bash and Harsch life test.

Metallurgical Analysis: Textbook

See abstract on p. 62.

Electropolishing of Nimonic 80

K. F. LORKING: 'The Electropolishing of Nimonic 80.' *Commonwealth of Australia, Dept. of Supply, Research and Development Branch, Aeronautical Research Laboratories: Structures and Materials Note* 205, May, 1953; 9 pp. + figures.

Following experiments with various baths, it was found that the best electropolishing results were obtained by using a solution containing 15 per cent. of sulphuric acid, 64 per cent. of orthophosphoric acid, balance water. Further investigation was directed to a determination of the influence of the following variables:—

(1) *Effect of Addition Agents.* Although certain organic compounds, such as glycerol, and surface-active agents, such as 'Teepol', have been found to improve the surface finish on copper electropolished in phosphoric-acid solutions, the addition of such agents to the phosphoric-sulphuric acid solution used for treatment of the Nimonic alloy caused a deterioration in the quality of the finish.

(2) *Current Density.* The current density giving optimum polishing corresponded to an applied voltage immediately below that at which evolution of oxygen began, but at such voltages some difficulty was experienced in stabilizing the polishing conditions. The results obtained indicated that it might be preferable to raise the temperature or stir the solution in order to ensure the conditions required.

(3) *Temperature of Operation.* The effect of bath temperature, over the range 20°-85°C., was studied. Raising the temperature was found to improve the stability of the bath conditions, and increase the rate of polishing. These modifications resulted in progressive improvement in the surface finish as the temperature was raised to 70°C., after which further rise caused deterioration.

(4) *Agitation of Polishing Solution.* The authors found no method of agitating the bath by which any improvement of surface finish could be produced.

In reviewing the results it is pointed out that the conditions required for electropolishing of Nimonic 80 are much more critical than those necessary for electropolishing of nickel. It is considered that the difference is due to the chromium in the alloy, since the presence of chromium ions would increase the range of stability and the packing density of the oxide film formed on the metal surface. The film so formed may, it is believed, be too thick or too densely packed for uniform dissolution of the metal surface, and it is necessary to find conditions such that the material of the film is readily soluble in the bulk of the solution. The beneficial influence of raising the temperature of the bath is discussed on theoretical grounds.

In the initial experiments in which bath composition was under study it was observed that polishing of Nimonic occurred to a certain extent in sulphuric-acid solutions at high current densities and in phosphoric-acid solutions over a wide range of current densities. The difference in the polishing characteristics of these baths cannot be readily explained in terms of current theories of the mechanism of the electropolishing process.

Hot Cracking in Welds made in High-Temperature Alloys

W. R. APBLETT and W. S. PELLINI: 'Factors which Influence Weld Hot Cracking.' *Welding Jnl.*, 1954, vol. 33, Feb., pp. 83S-90S.

This paper is concerned with (1) development of a test which will assess the liability of materials to hot cracking on welding, and (2) a study of the influence of relevant metallurgical and mechanical variables, on susceptibility to hot cracking.

As a basis to this work, casting studies had been made in the Naval Research Laboratory Washington, D.C., comprising an investigation (by thermal analysis and radiography), of the solidification of castings to determine the time of cracking. These had demonstrated that hot tearing occurs at temperatures slightly above the solidus, at which temperatures the metal consists of dendrite structures separated by essentially continuous films of liquid. Tensile tests of solidifying castings demonstrated that the metal does not develop measurable strength until the surface skin approaches the solidus temperature, and that an abrupt rise in strength of several pounds per square inch occurs on passing through the solidus. At temperatures slightly above the solidus the fractures are of 'square' type, exhibiting no evidence of ductility, and their appearance is similar to that of hot cracks. On passing through the solidus, however, the metal develops a high degree of ductility (as evidenced by large reduction of area prior to fracture), and the appearance of the fracture changes to a ragged fibrous type. A schematic drawing in the present paper illustrates the mechanism of hot-crack formation as deduced from the casting studies.

It was concluded from these tests that metallurgical factors which influence solidification conditions provide the conditions necessary for hot cracking, but that its actual occurrence is determined by the severity of the imposed-strain conditions. It is suggested that a similar inter-relation of metallurgical and mechanical factors exists in the welding of turbine blades to the rim of the wheels: conditions present in this operation are schematically illustrated, showing the various stages of solidification of the metal after the welding operation and the incidence of a stage at which conditions are favourable to hot cracking.

The turbine-blade attachment conditions were selected as those requiring consideration in a hot-cracking test which should embody the required combination of mechanical and metallurgical factors conducive to cracking. The test set-up developed (illustrated and described in the paper) provides for varying

Table I

Alloy	C %	Mn %	Si %	Cr %	Ni %	Co %	Mo %	W %	Nb %	Ti %	Fe %
N–155	0·17	1·67	0·66	21·0	20·5	30·2	—	—	—	—	Bal.
HU	0·66	0·67	1·52	17·2	40·0	0·27	0·12	—	0·22	—	Bal.
HW	0·48	1·13	1·43	17·0	60·3	0·23	0·08	—	0·22	—	Bal.
S–590	0·46	1·20	0·40	20·0	18·7	4·87	1·22	2·20	7·34	—	Bal.
EME	0·15	0·52	0·57	19·5	12·2	—	—	3·15	1·15	—	Bal.
16–25–6	0·09	1·68	0·63	16·3	25·2	—	6·25	—	—	—	Bal.
19–9 DL	0·29	1·00	0·82	19·2	9·0	—	1·34	—	—	—	Bal.
Inconel 'X'	0·05	0·48	0·48	14·5	74·5	—	—	—	0·93	2·3	6·8

Table II

Alloy	Liquidus		Solidus	
	°F.	°C.	°F.	°C.
N–155	2516	1379	2125	1163
HU	2489	1364	2315	1267
HW	2467	1351	2290	1253
S–590	2435	1334	2380	1303
EME	2600	1427	2428	1330
16–25–6	2548	1397	2440	1337
19–9 DL ..	2610	1432	2425	1328
Inconel 'X' ..	2498	1370	2395	1311

the degree of straining to which the solidifying metal is subjected, thus assessing liability of the material to hot cracking.

Test welds were made by the inert-gas-shielded tungsten-arc method in a number of alloys selected as representative of materials currently used for high-temperature applications. Their compositions are given in Table I above and liquidus and solidus temperatures are shown in Table II.

The extent of hot cracking, as a function of the severity of the mechanical stress conditions imposed in the test (shown graphically in the paper), indicates that N.155 alloy (which has the widest solidification range and the lowest solidus temperature) is the most susceptible to hot cracking: that intermediate cracking tendency is shown by alloys HW and HU type (having intermediate solidus temperatures and relatively wide solidification ranges), and that the remaining alloys (having high solidus temperatures) are characterized by low tendency to cracking. Within the least susceptible group the relatively small differences do not appear to bear a fixed relationship to solidification range: it is believed that there are other factors, possibly related to the type of dendrite structures formed, which influence hot cracking. The structure of the metal in the immediate proximity of hot cracks is illustrated for typical alloys in the series examined.

In a later stage of the investigation a study was made of the effects of large grain size in the base metal, and of the presence of low-melting-point segregates, on tendency to hot cracking. Experiments were made on coarse- and on fine-grained material containing abnormally high amounts of low-melting segregates. Fusion welds deposited on a coarse-grained sample of cast 18-8 and 19-9-Nb chromium-nickel steels containing about 0·10 per cent. sulphur developed cracks, but fine-grained material from the same castings and coarse-grained materials which had been hot-forged did not show cracks. The reasons for this difference in behaviour are discussed.

Metal-Refractory Materials

A. CARTER: 'Sintered Refractory Alloys.'
Metallurgia, 1954, vol. 49, Jan., pp. 8-14.

This is a survey of the present position of development of the metal-refractory materials variously known as cermets, cermals, and metamics. The author

indicates, in passing, the inaccuracy of the nomenclature which has been applied to certain of these types.

The general physical and mechanical properties characteristic of such materials, and some of their uses, are briefly discussed, together with a review of the methods of production employed for making the metal-refractory masses (cold pressing, followed by sintering; hot pressing and sintering; impregnation or infiltration of a refractory skeleton with the metallic constituent).

A detailed account is then given (supported by references to the literature) of the composition and properties of the various types of refractory metal composites. The most important group is that based on the carbides, especially titanium carbide. Outstanding in this group are materials comprising a solid solution of chromium carbide in titanium carbide, bonded by nickel-chromium, cobalt-chromium, or nickel-cobalt-chromium alloy: some notes are given on their properties, with particular reference to the influence of the amount of binder incorporated. Reference is made also to alloys based on titanium, tantalum, and niobium carbides, alloys containing other carbides, and metal-nitride, -boride, -silicide, and -oxide alloys.

The properties of the major cermet materials are compared with those of Nimonic 90 (nickel-cobalt-chromium-base alloy), as being the best wrought gas-turbine blading alloy currently in large-scale use. Although some of the cermets compare favourably with high-temperature alloys with regard to stress-rupture properties, and have the advantage of lower specific gravity (which will be significant in rotor-blading applications), development has not yet reached a stage when cermets are suitable to replace conventional forged blading alloys in gas-turbine rotors. Tests made to date indicate that an important feature in such progress may be the development of modified methods of attaching the blading to the turbine disc. For ranges of service temperature envisaged in the immediate future the titanium-carbide-base materials appear to have the greatest interest, but at still higher temperatures, e.g., of the order of 1300°C. and above, the chromium-alumina type of cermet is likely to be more promising.

Evaluation of Resistance to Corrosion by Acids at Elevated Temperatures

R. F. MILLER, R. S. TRESEDER and A. WACHTER: 'Corrosion by Acids at High Temperatures.' *Corrosion*, 1954, vol. 10, Jan., pp. 7–12.

The authors describe a simple method for assessment of the resistance of metallic materials to corrosion by acids at elevated temperatures and pressures. A small strip specimen of the test material is placed in a heavy-walled glass tube containing a small volume of the selected corrosive liquid. Air in the tube may be replaced by another gas, or evacuated. The tube is then flame-sealed, and placed in a vertical position in a constant-temperature oven for the desired test period. The pressure in the tube reaches the equilibrium value for the test temperature. The size of specimen usually employed is 0·125 in. × 0·25 in. × 2·0 in., and for a single specimen no support is needed. When it is required that the specimen shall be exposed to the vapour phase or to both vapour and liquid phases, simple glass supports can be used. The usual weight-loss and pit-depth measurements are made, and if stress-corrosion susceptibility is to be evaluated small stressed specimens of the U-bend type can be employed. Constant-load beam specimens have also been used. The apparatus is illustrated by diagrams, and test procedure is fully described.

The principal advantage of the test is its simplicity, and the large number of tests which can simultaneously be made. The use of all-glass apparatus is also obviously convenient. One of the limitations of such procedure is the absence of mechanical agitation, and a further one is the small amount of liquid which can be used.

If the corrosive constituent is gaseous, pressure control, and therefore concentration control, may be difficult. The method has, however, proved to be of considerable value as a screening test for making a quick survey of the types of material likely to offer good resistance under specific conditions.

Data demonstrating its usefulness are given in a series of tables showing the behaviour of series of metals and alloys in contact with the following corrodents: 5 per cent. sulphuric acid containing a small amount of ethyl alcohol, at 220°C. and 250°C.; 85 per cent. phosphoric acid, at 250°C.; nitric acid containing traces of various organic acids, at 150°, 175°, and 200°C.; boric acid (10–70 per cent.), at 120°–210°C.: 5 per cent. formic acid, at 170°C.; phenol containing hydrogen chloride and water, at 192°C. The materials exposed in all or some of the tests included six grades of straight or complex austenitic chromium-nickel steel, three high-chromium stainless steels, nickel, Monel, Inconel, Hastelloy B and C, Illium R, carbon steel, aluminium alloy, copper, Everdur, lead, platinum, gold, silver, tantalum, titanium, and silicon-iron alloy.

Resistance of Nickel-containing Cast Irons to Soil Corrosion

I. A. DENISON and M. ROMANOFF: 'Corrosion of Nickel Cast Irons in Soils.' *Jnl. of Research, Nat. Bur. Standards*, 1953, vol. 51, Dec., pp. 313–20; R.P. 2459.

This report summarizes results of tests made on nickel-containing cast irons of various types buried, for periods varying up to 14 years, at test sites having widely differing types of soil. The materials exposed contained (*a*) nickel in small percentages, with small amounts of copper, (*b*) nickel about 3 per cent., without copper, and (*c*) higher amounts of alloys, *viz.* nickel 15, copper 6·58, chromium 2·61, per cent. Plain cast iron was exposed for comparison. Most of the material was in the form of piping; two of the irons were exposed as plate. The soils were representative of inorganic oxidizing, inorganic reducing, and organic reducing types, and at one site the exposure was in cinders.

Evaluation of resistance to corrosion was by measurement of weight loss, and of maximum depth of pitting, and determination of hydraulic bursting pressure.

The results of these tests showed that additions of nickel up to 3 per cent. significantly reduced the initial corrosion of cast iron in poorly drained soils of low resistivity, but that this advantage was not maintained for the full duration of the exposures. The high-alloy cast iron showed good resistance to corrosion in most of the soils, especially as assessed by maximum depth of pitting. With some exceptions, samples of severely corroded plain and low-alloy cast iron pipe withstood a maximum hydraulic pressure of 500 p.s.i. after exposure at the test sites for periods up to 11 years and storage in the laboratory for about a year. In view of the effect of oxidation in air, in weakening the layer of corrosion products, the observed values for hydraulic bursting pressure are probably to be considered as minima.

Production of Stainless Steel Aft Frames for Aircraft Gas Turbines

L. M. LIMBACH: 'The Production of Aft Frames for Aircraft Gas Turbines.' *Machinery (Lond.)*, 1954, vol. 84, Feb. 5, pp. 273–8.

An important component of an aircraft gas turbine is the aft frame, which holds the combustion chambers, transition liners, turbine casing, and compressor casing, in one assembled unit. The aft frame also contains the housings for two of the main bearings of the high-speed turbine compressor shaft.

Aft frames for the General Electric J-47 jet-propulsion units are fabricated throughout from 18-8 chromium-nickel steel, and are machined to tolerances which are unusually close for a structural part. This article gives a detailed account, with extensive illustrations, of the machining equipment and methods employed by Ryan Aeronautical Company, in whose works these units are produced.

The aft frame is in the form of a slightly tapered cylinder, to which are welded several heavy-gauge machined flanges and a large circular burner plate. Eight flanged eyelets in the burner plate receive the combustion chambers, and hold them in accurate alignment. More than 150 feet of high-quality welding is required in fabricating each frame: electric welding is employed in making the tapered cylinder, and metallic arc welding for attaching the flanges to the barrel and for joining other parts. The aft frames are considered to be among the most complex stainless-steel products constructed in the Ryan plant, which has for many years specialized in the fabrication of welded components for the aircraft industry. On completion of welding, and prior to undergoing the machining operations described in the present article, the frames are heat-treated in a large controlled-atmosphere electric furnace, for removal of residual strains.

Descaling of Stainless Steels

L. F. SPENCER: 'Descaling the Stainless Steels.' *Metal Finishing*, 1954, vol. 52, Feb., pp. 54–9.

After a brief discussion of mechanical descaling (which was more fully dealt with in an earlier article, *ibid.*, 1953, vol. 51, Mar., p. 70), the author describes at length procedures which may be used for acid pickling or descaling with caustic. Details are given of seven acid pickling baths which can be recommended, under various conditions, for pickling of chromium-nickel and/or chromium stainless steels. The advantages and limitations of each type are critically considered. Mention is made of the increasing popularity of caustic descaling baths, and the sodium hydride process and another fused bath are described as typical.

Friction Sawing of Nickel-containing Materials

See abstract on p. 62.

Corrosion-Resisting Nickel Alloys in the Paper-making Industry

M. A. SCHEIL: 'Hastelloy Alloy F. A New Corrosion-Resistant Alloy for Lined Digesters.' *TAPPI*, 1953, vol. 36, June, pp. 241–7.

This article summarizes information on the corrosion-resisting alloy developed some three years ago by Haynes Stellite Company: see earlier references in *Nickel Bulletin*, 1953, vol. 26, Nos. 5 and 7, pp. 87 and 120. The composition limits are given as carbon 0·08 max., manganese 1–2, phosphorus 0·040 max., sulphur 0·030 max., silicon 1·00 max., chromium 21–23, nickel 44–47, molybdenum 5·5–7·5, niobium + tantalum 1·75–2·5, tungsten 1·00 max., cobalt 2·50 max., copper 0·15 max., per cent., iron balance. The information given with regard to corrosion-resistance and mechanical and physical properties is similar to that summarized in *Nickel Bulletin*, 1953, vol. 26, No. 7, pp. 120–1.

The most important application of the alloy to date has been in lined digesters used for preparing sulphite pulp: this article is based mainly on experience of such use, in both acid and in alkaline digesters. Welding characteristics of the alloy are discussed in some detail, in relation to the use of Hastelloy-lined equipment in various types of chemical plant.

Welding of Inconel X

E. F. NIPPES and H. B. FISHMAN: 'Optimum Spot and Seam Welding Conditions for Inconel X.' *Welding Jnl.*, 1954, vol. 33, Jan., pp. 1S–14S.

This report covers one section of the research on resistance welding of nickel and high-nickel alloys which has been continuously in progress at Rensselaer Polytechnic Institute since 1939. References are given to earlier reports on welding conditions for nickel, Inconel, and Monel.

The aim of the work now reported was the determination of the optimum spot- and seam-welding conditions for Inconel-X sheet in thicknesses of 0·010, 0·015, 0·021, 0·031 and 0·062 in., optimum conditions being taken as signifying those satisfying the criteria laid down in the *Resistance Welding Manual* of the Resistance Welding Manufacturers' Association, viz.:

(1) No porosity or cracking in the fusion zone.
(2) Less than 5 per cent. indentation or 10 per cent. sheet separation.
(3) Fusion-zone penetration of 30–80 per cent. of the total thickness of the sheet.

Gauge	Heat	C %	Mn %	Fe %	S %	Si %	Cu %	Ni %	Al %	Cr %	Ti %	Nb %
0·010 0·015	Y3576X	0·05	0·54	6·75	0·007	0·41	0·03	73·59	0·78	14·35	2·43	1·04
0·021	Y3400X	0·04	0·40	6·17	0·007	0·41	0·01	74·34	0·71	14·39	2·48	1·01
0·031	Y3402X	0·04	0·42	6·58	0·007	0·41	0·02	73·46	0·64	14·89	2·54	1·00
0·062	Y3401X	0·04	0·41	6·42	0·007	0·33	0·01	73·78	0·69	14·72	2·48	1·05

(4) 10–25 per cent. fusion-zone overlap for pressure-tight seam welds.
(5) A wide and reproducible current range. (The welding current was limited at the low end by insufficient fusion, and at the high end by expulsion or surface spitting.)

In addition to non-destructive and metallographic tests made on each group of welds, tension-shear and cross-tension tests were made on both as-welded and welded and age-hardened material. Physical testing for pressure-tight seam welds was carried out by means of pillow tests.

Compositions of the sheet used are given in the table above.

The sheet was cold-rolled to size and solution treated, to leave it in the soft-tempered condition for welding.

The most significant properties of Inconel X are its high tensile, creep, and stress-rupture strengths in the temperature range 1000°–1500°F. (540°–815°C.). Various combinations of properties within that range can be secured by suitable heat-treatments: typical values for the alloy in various conditions are quoted in this paper.

A detailed report is made on the equipment used, tests applied, and properties obtained in the welded materials. The welding variables investigated, as affecting the quality of the joints producible, included geometry of the electrode, welding current, weld force, weld spacing, and welding time.

The extensive experimental work carried out indicated broadly that no major problems should be encountered in spot or seam welding of this material provided that the welding conditions laid down as optimum are maintained, and that the stock (before welding) is given a uniform surface treatment to remove the surface oxide film. Optimum weld force was found to be a linear function of sheet thickness. Consistency in strength of spot welds may be improved by increasing the weld time, but care must be taken not to exceed sheet-separation or indentation limits. The fused diameter or width should be sufficiently large to cause plug failure when 30 per cent. penetration is attained. Increasing the fusion-zone overlap reduces the tendency to expulsion, and so broadens the optimum current range permissible during seam welding. In the pillow test it was found that maximum rupture strength is a linear function of thickness of sheet. The results obtained in this research gave no indication that coring was detrimental to the mechanical properties of the welds.

Determination of Rare Earths in Stainless Steels

E. W. SPITZ, J. R. SIMMLER, B. D. FIELD, K. H. ROBERTS and S. M. TUTHILL: 'Spectrographic Determination of Certain Rare Earths in Stainless Steels.' *Analytical Chemistry*, 1954, vol. 26, Feb., pp. 304–7.

The addition of rare-earth oxides or metals to molten steel, for improvement of mechanical and physical properties, has stimulated interest in development of relevant methods of analysis. This paper describes procedure based on (1) electrolytic removal, at the mercury cathode, of the major steel components, including chromium, followed by (2) chemical treatment to remove as much as possible of impurities such as calcium and aluminium which remain in the rare-earth concentrate, and (3) spectrographic determination of cerium, lanthanum, neodymium and praseodymium by means of a direct-current arc, under conditions which eliminate interference by small amounts of elements not removed by either the electrolytic or the chemical separation step. Results are given of typical determinations made by this method, on various austenitic chromium-nickel steels.

Stainless-Steel Powders Produced by Water Disintegration

W. L. BATTEN: 'Powdered Metals Enter Stainless Stage.' *Steel*, 1953, vol. 133, Dec. 1, pp. 78–81.

The article describes the properties and uses of stainless-steel powders made by water-disintegration of pre-alloyed molten metal. Steel of the desired composition is melted in induction furnaces and is transferred to a tundish lined with zirconium-silicate refractory and heated electrically to control and maintain the temperature of the molten metal. The liquid metal flows through a small zirconia nozzle (hole diameter $\frac{3}{16}$–$\frac{7}{32}$ in.) into the disintegrator, where the thin stream is blasted to powder by water jets emerging from a hollow rapidly rotating disc. The disc is attached to the top of a hollow shaft through which water flows at low pressure, and the centrifugal action of the revolving disc builds up pressure sufficiently high to supply the required disintegrating energy. (A schematic drawing of the equipment is shown.) The mixture of water and powder is then pumped to a dewatering and classifying unit, in which sorting of powder according to particle size, and final annealing are carried out.

This article gives information on properties typical

of stainless-steel powders of various compositions and sizes made by this process, and of compacts prepared from the powders. Several of the standard austenitic nickel-chromium steel grades are represented, as well as steels of lower alloy content.

Among the applications for which parts made from these powders have been successfully used are small driving arms in intermediate gear trains of water meters, sleeve bearings and bushings working under corrosive conditions, guides for fishing rods, small gears, and levers. Filters are also an important application of stainless-steel powder, and other uses include porous sheets used in de-icing equipment, and spray coatings.

PATENTS

Surface Treatment of Metallic Materials Prior to Rolling

Surface imperfections on metals, and on many types of steel and nickel-base and other alloys, are converted to a loose, easily removable scale by heating the material at 700°–1200°C. in an atmosphere of a gaseous sulphur compound, preferably hydrogen sulphide.

W. O. BINDER, assignor to UNION CARBIDE AND CARBON CORPN. *U.S. Pat.* 2,637,667.

Production of Nickel-Alkaline Batteries

Blistering of the plates of alkaline accumulators made from sintered carbonyl nickel powder impregnated with nickel oxide or cadmium oxide is prevented by keeping the temperature of the plates below 200°F. (93°C.), and preferably below 135°F. (55°C.) during the assembly of the cell, e.g., by welding.

M. J. STUMBOCK. *U.S. Pat.* 2,667,526.

Electrodes for secondary batteries are made by sintering onto a wire-mesh base a metal powder (e.g., copper-nickel powder mixture on an iron mesh). The mesh is then nickel-plated, and the porous sintered plate (preferably of nickel) which will carry the active material is sintered on. The powder applied to the mesh should be such as to form, on sintering, an alloy which protects the mesh from electrochemical attack, and should roughen it so that the porous plate sinters firmly on.

ACCUMULATOREN-FABRIK, A.G. (inventor, P. HAGSPIHL). *Brit. Pat.* 701,859.

Regeneration of Nickel Sulphide Catalysts

A partially spent catalyst comprising nickel sulphide associated with activated alumina or amorphous alumina, which has been used in reduction of an aromatic nitro compound with hydrogen, is regenerated by treatment with a gas containing free oxygen, at about 300°–700°C. Treatment is continued long enough substantially to remove carbonaceous impurities present on the catalyst, and subsequently, without any intervening sulphiding step, the catalyst thus oxidized is treated with a gas containing hydrogen, at about 200°–700°C.

ALLIED CHEMICAL AND DYE CORPN. *Brit. Pat.* 702,018.

Stabilization of Nickel-containing Soaps

Metal soaps, including cobalt and nickel soaps, which are used as paint driers, are stabilized by mixing with them a salt of an organo-substituted acid of phosphorus. Where the salt is that of a drier metal, the soap may be omitted from the drier composition.

J. C. ARNOLD, communication from FERRO ENAMEL CORPN. *Brit. Pat.* 704,333.

Nickel and Nickel Alloy Additions to Paints

A coating composition, e.g., paint, is rendered electrically conductive by incorporating 5 to 25 per cent. by volume of a non-oxidized magnetic metal powder, preferably nickel, soft iron, magnetic steel, Permalloy, or Alnico.

IMPERIAL CHEMICAL INDUSTRIES, LTD. (inventor, A. J. ELLEMAN.) *Brit. Pat.* 705,166.

Production of Nickel Powder with Large Surface Area

Nickel oxalate is heated at 200°C. until all the water of crystallization is driven off, and the anhydrous salt thus formed is heated *in vacuo* at 310°–330°C. until decomposition is 99–100 per cent. complete. It is claimed that powders having surface areas of 54 m^2/gm. are so obtained.

N. J. DE LOLLIS, assignor to U.S. SECRETARY OF COMMERCE. *U.S. Pat.* 2,660,523.

Electrodeposition of Bright Nickel Coatings

A bright nickel coating claimed to be highly ductile is electrodeposited from a bath containing essentially nickel, hydrofluoric acid, boric acid, and water, plus a brightener. The hydrofluoric acid reacts with the nickel, to form a complex nickel fluoride, and is present in a quantity greater than that required to convert the nickel to the complex nickel fluoride, with the result that the excess hydrofluoric acid gives the bath a pH of more than 1 and less than 6. The anions of the bath comprise essentially fluorides and borates, and the bath contains at least 30 grams of nickel metal per litre. One or more brightening agents is present (preferably a mixture of paratoluene sulphonamide, saccharin and fuchsin), but such addition does not interfere with the ductility of the deposit.

INTERNATIONAL KENMORE, LTD., E. L. A. CAMIN, S. E. HYBINETTE and H. B. TAYLOR. *Brit. Pat.* 701,717.

Bright electrodeposits are rapidly obtained by the use (in a sulphate or sulphate-chloride bath) of a single brightening agent of the bis-pyridinium type or a mixture of equivalent addition agents of the same type. Numerous suitable compounds are specifically mentioned.

J. D. LITTLE, assignor to HARSHAW CHEMICAL CO. *U.S. Pat.* 2,658,867.

Bright, ductile nickel electrodeposits are obtained from a bath containing nickel fluoborate and small

quantities of sulphurized quinoidine and benzoic sulphimide.
N. F. BLACKBURN, assignor to PENNSYLVANIA SALT MANUFACTURING CO. *U.S. Pat.* 2,664,392.

Unsaturated polyvalent aliphatic alcohols, particularly tertiary alcohols, and preferably dimethylacetylene-carbinol, are added as brightening agents, to nickel-plating baths, in amounts of 0·1–1 g./L.
W. KAMPSCHULTE AND CO. *French Pat.* 1,032,922.

Fine-grained bright electrodeposits of nickel, cobalt, or their alloys are obtained from a bath comprising an aqueous acid solution of the chloride, sulphate or fluoborate of the metal to be deposited (or two or more of these salts) and containing also an organic sulphinate. Aryl sulphinates, particularly toluene sulphinate, are preferred. They may be used in low concentration, in combination with other brightening agents, e.g., with O-benzoyl sulphimide.
H. BROWN, assignor to UDYLITE CORPN.
U. S. Pat. 2,654,703.

A brightening agent for use in the electrodeposition of nickel from an aqueous acid bath consists of the product resulting from reduction of a triphenylmethane dye by means of zinc powder and hydrochloric acid in the presence of sodium bisulphite or sodium hydrosulphite. Suitable reducible dyes are: fuchsin, pararosaniline, rosaniline, methyl violet, aniline blue, iodine green, aurine, acid fuchsin.
L. B. PORTZER and W. C. LEITENBERGER.
U.S. Pat. 2,637,687.

Nickel Plating of Electric Discharge-Tube Parts

A relatively thick layer of nickel or copper is applied, electrolytically or by spraying, to the metal exhaust tubes of electric discharge tubes, to strengthen them before closure by mechanical pinching.
PHILIPS ELECTRICAL INDUSTRIES, LTD.
Brit. Pat. 704,408.

Chemical Process for Deposition of Nickel

Copper, silver, gold, aluminium, iron, nickel, palladium or platinum may be chemically coated with nickel by immersion in an aqueous solution containing a nickel salt, a hypophosphite, and a salt of a simple short-chain saturated aliphatic dicarboxylic acid. Sodium succinate and malonate are preferred.
G. GUTZEIT and E. J. RAMIREZ, assignors to GENERAL AMERICAN TRANSPORTATION CORPN.
U.S. Pat. 2,648,842: see also *Belgian Pat.* 521,467.

A related patent claims a continuous process of chemical nickel plating, using the baths covered by *U.S. Pat.* 2,658,842.
P. TALMEY and W. J. CREHAN, assignors to GENERAL AMERICAN TRANSPORTATION CORPN.
U.S. Pat. 2,658,839: see also *Belgian Pat.* 521,551.

Nickel-Coating of Non-Conducting Materials

In metallizing non-conducting materials, including non-metallics, an adherent nickel film is formed by using a nickel-pyridine complex and employing sodium hydrosulphite as the reducing agent. The non-conductor is preferably sensitized prior to plating, using, for example, tin chloride or hydrochloric acid for glass or zinc chloride for plastics.
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, INDIA (inventors, T. BANERJEE and K. CHAKRABARTY).
Indian Pat. 45,579.

In a related patent, metallization is effected by formation of a primary conducting film of nickel from a nickel-cobalt complex, for chemical reduction in the presence of sodium hydrosulphite prior to electroplating.
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH (inventors, K. CHAKRABARTY, B. N. GHOSH and S. K. ROY). *U.S. Pat.* 45,695.

Coating of Aluminium with Nickel by Decomposition of Nickel Carbonyl

A firmly adherent coating of nickel is produced on aluminium by (1) applying a thin porous nickel film to the clean metal base by contacting it, heated to 375° to 400°F. (190° to 205°C.) with a stream of gaseous nickel carbonyl diluted with a non-oxidizing gas; (2) heating the base thus coated to 500°–1200°F. (260°–650°C.) while maintaining an atmosphere of carbon dioxide round the aluminium; and (3) exposing the nickel-coated base to nickel carbonyl gas while the base is heated to a temperature sufficiently high to decompose the gas, with resulting deposition of a coating of nickel of the desired thickness.
A. O. FINK, assignor to COMMONWEALTH ENGINEERING CO. *U.S. Pat.* 2,653,879.

Electrodeposition of Nickel-Tungsten Alloys

Alloys of tungsten with iron, cobalt, or nickel are electrodeposited from alkaline aqueous baths containing tungstate ions, a complex of ammonium ions, and ions of iron, nickel, or cobalt, also aliphatic organic ions having at least three –OH and $-CO_2H$ groups in total. Suitable organic ions are those of citric, tartaric, malic, and gluconic acids. The nickel-tungsten alloys covered by the patent may contain 1–40 per cent. tungsten.
A. BRENNER, P.S. BURKHEAD and C. A. SENTEL, assignors to U.S. SECRETARY OF WAR. *U.S. Pat.* 2,653,128.

Heat-Treatment of Nickel-Tungsten Alloys

Sintered nickel-tungsten alloys (5–45 per cent. nickel) are heated to 1000° to 1200°C., quenched, and reheated at 500°–700°C. Their mechanical strength is increased without appreciable hardening of the alloy.
C. VERDI, assignor to FANSTEEL METALLURGICAL CORPN.
U.S. Pat. 2,655,457.

Steel for Hot-Forging Dies

Steel for dies, characterized by high resistance to erosion under metal flow at high temperatures, and able to withstand repeated heating and cooling, contains carbon 0·05–0·80, manganese 0·20–1·20, silicon

0·10–1·50, nickel 2·00–5·00, molybdenum 2·00–4·50, per cent., iron balance. A suitable heat-treatment is described.

HEPPENSTALL CO. *French Pat.* 1,023,444.

Precipitation-Hardening Alloy for Use at High Temperatures

A ferrous material suitable for high-temperature applications, e.g., parts of gas turbines, contains chromium 15–25, nickel 25–35, titanium 2·4–6, carbon 0·5–0·75, per cent., iron balance, with not more than 0·02 per cent. of free or combined oxygen or nitrogen. The gaseous-impurity levels may be kept to a minimum by using the vacuum melting process described in *U.S. Pat.* 2,564,498. The effect of excluding oxygen and nitrogen, on strength and rupture life, is exemplified for typical alloys.

J. D. NISBET, assignor to GENERAL ELECTRIC CO. *U.S. Pat.* 2,661,284.

Precipitation-Hardenable Austenitic Steel

A precipitation-hardenable 18–8 type steel contains chromium 17–21, nickel 7–14, silicon 2–4, molybdenum 1·5–4, beryllium 0·05–0·20, carbon up to 0·12, per cent., iron balance. Corrosion-resistance may be improved by addition of 1–4 per cent. of copper.

N. S. MOTT, assignor to COOPER ALLOY FOUNDRY CO. *U.S. Pat.* 2,635,044.

High-Strength Austenitic Steels

Components of high yield strength are produced, without cold working, by subjecting to solution and precipitation-hardening treatment a steel containing carbon 0·35–0·55, manganese 3 to less than 5, nickel 10–13, chromium 2·5–5·5, vanadium 0·5–1, per cent., remainder iron with incidental impurities. The treatment comprises heating to 1100°–1200°C. for a suitable period, cooling in air, oil, or water, and immediately subjecting the material, without the intervention of cold working, to a heat-treatment at 650°–750°C. Typical periods of treatment are cited for specified temperatures.

C. SYKES, H. W. KIRKBY and C. STOKES. *Brit. Pat.* 703,636.

Complex Nickel-base Corrosion-Resisting Alloy

An alloy having high tensile strength and resistance to attack by acids, alkalis, sea water, and corrosive gases contains nickel 69–80, chromium 16–25, manganese 2–4, molybdenum 0·5–2, per cent. Corrosion-resistance and malleability may be enhanced by the addition of 2–6 per cent. of silver, and it is preferred that the alloys contain about 1 per cent. of aluminium and a small but effective percentage of boron.

M. GORSCHALKI. *U.S. Pat.* 2,661,285.

Processing of Precipitation-Hardenable High-Temperature Alloys

Complex nickel-chromium-base alloys of specified composition are formed into engineering parts by (*a*) shaping at elevated temperatures, (*b*) solution heat-treatment for 5–10 hours at 1050°–1200°C., (*c*) machining to finished size, (*d*) short-time heat-treatment at 1050°–1100°C., (*e*) precipitation heat-treatment at 700°–800°C. for 10–20 hours. The hot-working step (*a*) may be interrupted when 75–95 per cent. complete, being finished after the solution heating. In such case a refining heating of up to 1 hour at 850°–1050°C. may be given before machining (cf. *Brit. Pat.* 674,723). The process covered by the present patent is specially suitable for alloys covered by *Brit. Pat.* 666,399 (see *Nickel Bulletin*, 1952, vol. 25, No. 4, p. 119), and for alloys containing chromium 18–22, titanium 2·2–2·8, aluminium 0·3–1·2, per cent., balance nickel.

ROLLS ROYCE, LTD. (inventors, H. E. GRESHAM, M. A. WHEELER and A. DUNLOP.) *Brit. Pat.* 703,483.

Degasification of Austenitic Steels

A ladle treatment for reducing porosity and similar defects in austenitic steel comprises adding to the molten steel, in the ladle, a degasifying agent consisting of selenium and/or tellurium, in solid form and in an effective amount not greater than about 0·03 per cent. by weight of the steel.

R. J. WILCOX and F. G. VIHTELIC, assignors to EBALOY, INC. *U.S. Pat.* 2,661,279.

Refinement of Grain in Stainless and Heat-Resisting Steels

Fine grain size is produced in heat-resisting and stainless steels by treating the melt, after addition of the alloying elements, with a composition containing rare-earth metal oxide, and subsequently deoxidizing. The metal should be chill cast.

N. F. TISDALE and N. F. TISDALE, jnr., assignors to MOLYBDENUM CORPN. OF AMERICA. *U.S. Pat.* 2,643,949.

Annealing of Stainless Steel

Stabilized stainless steels may be annealed in a nitrogen-containing atmosphere (e.g., cracked ammonia) by adding to the atmosphere a small amount of an oxidizing agent, sufficient to maintain an oxide coating on the steel, even at the maximum temperature used in annealing. The preferred addition is water vapour.

ROBERTSHAW-FULTON CONTROLS CO. (inventors, C. D. BRANSON and R. A. SMITH). *Brit. Pat.* 702,837.

High-Nickel Alloy Welding Electrode

A welding electrode suitable for welding articles having nickel surfaces and/or for applying nickel overlays to ferrous surfaces comprises (1) a core consisting of nickel containing titanium 3·5–5, aluminium up to 0·75, carbon up to 0·1, magnesium up to 0·35, per cent., and (2) a covering consisting essentially of calcium carbonate and one or more fluorides.

MOND NICKEL CO., LTD. *Brit. Pat.* 702,697.

Flux for Nickel-Chromium-Iron Welding Rods

A nickel-chromium-iron welding rod for use with austenitic nickel-chromium steels is coated with a flux containing 0·25–25 per cent. by weight of elementary sulphur, which acts to retard carburization.

P. S. VILES, assignor to STANDARD OIL DEVELOPMENT CO. *U.S. Pat.* 2,639,362.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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